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ФИЗИКА КОНДЕНСИРОВАННОГО СОСТОЯНИЯ
PHYSICS OF THE CONDENSED MATTER

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A.U. Aldiyarov, K.I. Beisenov*, U.P. Suiinzhanova, D.E. Yerezhep

Al-Farabi Kazakh National University, Almaty, Kazakhstan
*(*E-mail: khasen.007@gmail.com)*

Study of polyamorphic transformations in the cryomatrix of nitrogen in cryovacuum condensates of water

One of the important tasks of modern physics of condensed matter is to establish an unambiguous connection between the conditions of formation and the properties of the resulting solid phase. Its solution will contribute to major breakthroughs in the creation of materials with desired properties. As any scientific and technological problem, this approach is associated with the need to address a wide range of fundamental issues. The basis for success in this direction is the implementation of a complex not only with model tests, when the investigated substance is important from a practical point of view, but in itself has interesting physical properties; such objects can be fully attributed to chemical properties. Hydrogen-bonded substances, in which, in addition to van der Waals forces, interactions due to the presence of an intermolecular hydrogen bond play an important role. The obtained method of cryomatrix isolation facilitates assuming that in the process of cryocondensation of pure components of water and ethanol at an intermediate stage in the adsorbed layer, there is a process of formation of clusters with a short-range order similar to the liquid state of water or ethanol.

Keywords: crystallization, mixes, glass transition dynamics, crystal cell.

Introduction

The structure of water crystals is often determined by the presence of hydrogen bonds. This is due to the fact that the water molecule is a symmetric proton donor and acceptor. This distinguishes the water molecule from isoelectronic homologues such as NH_3 and HF. The NH_3 molecule has three protons and one pair, and the HF molecule has one proton and three single pairs. Thus, only in the system of H_2O molecules, hydrogen bonds (H-bonds) completely determine the geometry of H_2O crystals and the properties of condensed water. This is determined by the strong orientation of the H-bond, which means that if a hydrogen atom is between two oxygen atoms, then the spatial organization of such a system cannot be arbitrary. The formation of one hydrogen bond leads to a decrease in the activation barrier for the formation of the next H-bond, and so on. Since this cooperative property of hydrogen bonds is due to the interaction of two with hydrogen, one molecule is acidic and the other is alkaline. In this regard, it seems necessary to pay more attention to the structure of the water molecule [1, 2].

The structure of the water molecule. H_2O molecule consists of two hydrogen atoms and one oxygen atom. When studying the optical spectra of water, it was found that in the absence of motion (without oscillations and rotations), hydrogen and oxygen ions should be located on the vertices of a right triangle with an angle of 104.5°. The nuclei of a water molecule are surrounded by an electron cloud with a radius of 0.138 nm, consisting of positive electrons that are unevenly distributed within the sphere. Two of them are in the first orbit, in the immediate vicinity of the oxygen nucleus and do not play a significant role in the formation of the bond between oxygen and hydrogen, the remaining eight electrons are paired in four eccentric orbits in the tetrahedral direction from the oxygen nucleus. The charge of the eight electrons completely compensates for the charge of the oxygen nucleus, but the electrons rotating in two orbitals without protons form two negative centers, that is, the single electrons form two arms from the oxygen nucleus to the vertices of the imaginary tetrahedron, the H_2O molecule. The interval between H^+ and O^2 ions in the unexcited state is 0.96 $\overset{\circ}{A}$. Due to this structure, the water molecule is a dipole, because the density of electrons in the region of O^2 — ions is much higher than in the region of H^+ ions. One can imagine two small bulging water molecules in the region of the protons, as illustrated in Figure 1.

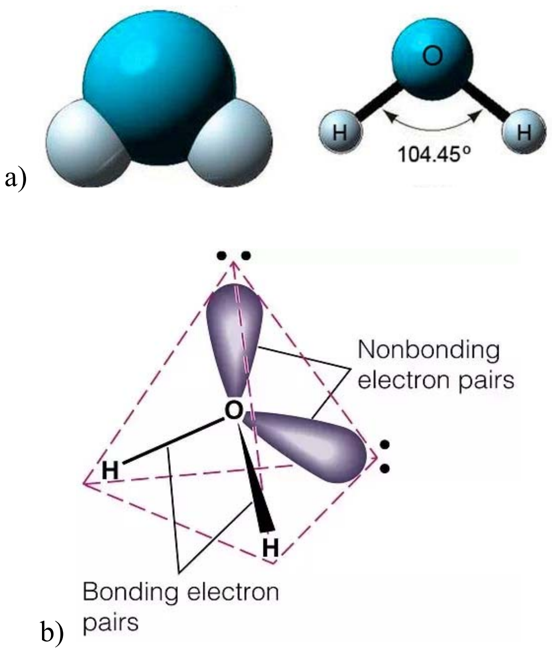


Figure 1. Geometric diagram of the monomer H_2O (a), plane model (b)

Dynamics of water molecules. As in a rigid structure, the nuclei of molecules in a crystal lattice are in a state of continuous oscillation at 0 K. An important feature of these oscillations is that they can be characterized by a limited number of fundamental oscillations, called normal modes. This is an oscillation in which all the nuclei oscillate at the same frequency and in the same phase. The water molecule has three normal modes ν_1, ν_2, ν_3. Any possible vibration of this molecule can be described as a superposition of these three modes.

The oscillations that move the H-nuclei in the direction of the OH bond are called the OH bond oscillations as shown in Figure 2. These oscillations occur at frequencies ν_1 and ν_3. The oscillations in which the H nuclei move in a direction almost perpendicular to the O–H bonds (ν_2 mode), are called the deformation vibrations of the H–O–H bonds or the bending oscillations of the H–O–H bonds. In fact, in the ν_1 mode, the H–O–H bond has a small amount of bending, while in the ν_2 mode, a small amount of O–H elongation corresponds. ν_3 is called asymmetric tensile vibration or asymmetric tensile vibration or asymmetric tensile vibration, and is different from ν_1 symmetric tensile vibration. These frequencies are derived from Raman and infrared spectra [3].

The transition of the water molecule from the basic state of oscillation, described by the mode ν_2, to the excitation corresponds to the infrared absorption band 1595 cm^{-1}. During this transition ν_2 describing the mode υ_2 quantum number varies from 0 to 1, and υ_1 and υ_3 describe the modes, ν_1 and ν_3 are zero quantum numbers. Similarly, the transition of a water molecule from the basic state of oscillation to the state in which only the first normal mode is moving (the state in which the quantum numbers $\upsilon_1=1$, $\upsilon_2=0$, and $\upsilon_3=0$) corresponds to an absorption band of 3657 cm^{-1}. The third normal mode corresponds to an absorption band with a maximum frequency of 3756 cm^{-1}. The given parameters correspond to an empty water molecule. During the transition to the condensed state, there are changes in the parameters of intermolecular interactions, as well as a significant expansion of the absorption bands of fundamental frequencies as a result of the formation of hydrogen bonds and their transition to lower vibration frequencies. This leads, in particular, to the superposition of the elongated vibration ranges, so that in the condensed states these frequencies are not separated, but are appeared as a single absorption band [4–5].

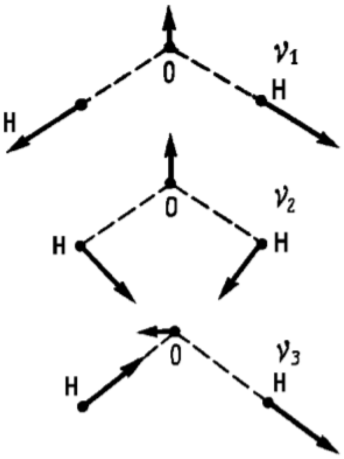
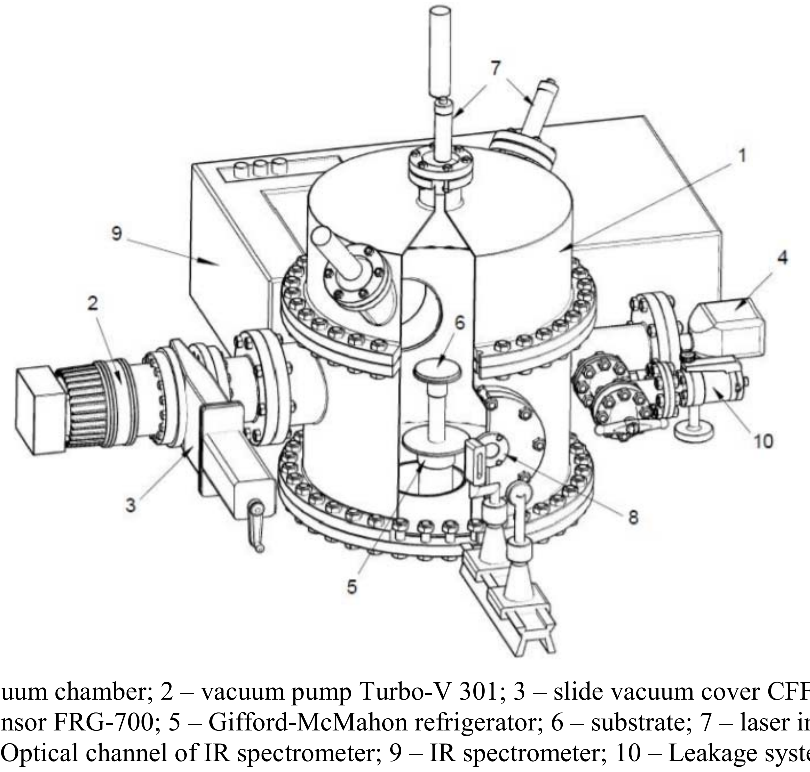


Figure 2. Normal oscillation modes of water molecules

Experimental

When studying the structural and phase transformations in the cryovacuum-condensate in the technological process, it is important to have information about the growth rate, refractive index and reflective characteristics of the cryocondensate-substrate system with a wide frequency range from 4200 to 400 cm^{-1}. To carry out this type of research, the existing experimental equipment was used in the Laboratory of Cryophysics and Cryotechnology of the Faculty of Physics and Technology. In addition, the need for low-temperature measurements and the study of cryocondensation films in the thickness range of 25–30 μm has led to an increase in requirements for both the functional capabilities of a number of important units and its technological parameters. These requirements relate to the maximum vacuum in the operating chamber of the unit, as well as the need to measure in the average IR range (400 cm^{-1}) [6–7].

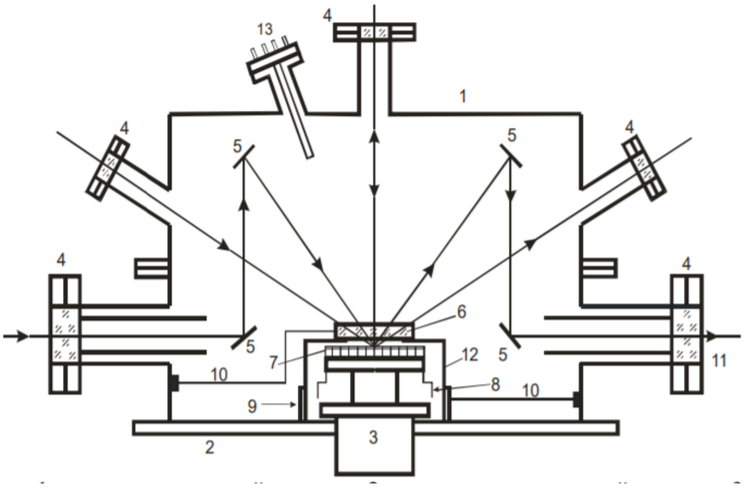


1 – vacuum chamber; 2 – vacuum pump Turbo-V 301; 3 – slide vacuum cover CFF–100;
4 – pressure sensor FRG-700; 5 – Gifford-McMahon refrigerator; 6 – substrate; 7 – laser interferometer;
8 – Optical channel of IR spectrometer; 9 – IR spectrometer; 10 – Leakage system

Figure 3. Schematic of the experimental unit

Figure 3 shows the schematic diagram of a universal vacuum cryogenic spectrophotometer in which a water cryocondensate is obtained in a vacuum chamber. The main installation unit is a cylindrical vacuum chamber with a diameter and height of 450 mm (1). The evacuation of the vacuum chamber was carried out by a turbo-molecular pump (2) Turbo-V 301 connected to the chamber of the CFF-100 sliding gate valve (3). The SH-110 dry circulation pump (not shown) is used as a forevacuum pump). The final vacuum in the chamber reached a value of not less than $P=10^{-8}$ Topp ($P=10^{-6}$ Pa). The pressure in the chamber was measured with a wide-range pressure sensor with FRG-700 controller.

In the center of the chamber is the Gifford-McMahon microcryogenic system (5), on the upper flange of which is mounted a mirror substrate (6), which serves as a surface for ethanol condensation. The substrate is made of copper, its working surface is covered with silver. The diameter of the substrate is d=60 mm. The minimum condensation temperature is T=16K. The temperature was measured with a DT 670–1.4 silicon sensor and a M335/20s temperature controller. Thickness and condensation rate are measured with a two-beam laser interferometer based on P25a-SS-0–100 — photomultiplier tubes (7). The absorption spectra were measured in the frequency range 400 cm^{-1}÷4200 cm^{-1}. Through ADC, the signals of the photomultiplier are sent to the computer and controlled by the PowerGraph program [8].



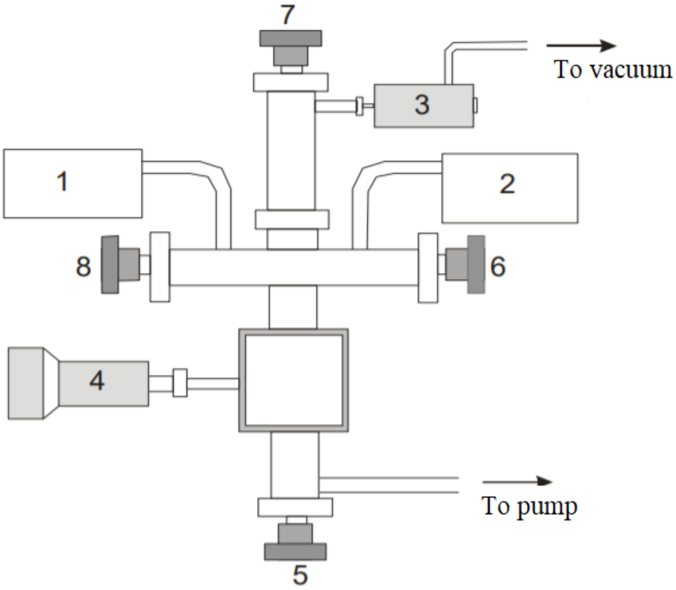
1 — vacuum chamber housing; 2 — the base of the vacuum chamber; 3 — McMahon micro-refrigerator;
4 — optical windows; 5 — light guides; 6 — protective plates KBr; 7 — substrate; 8 — cryopump;
9 — damper; 10 — magnetic drive; 11 — optical window KBr;

Figure 4. Schematic of the vacuum chamber of the spectrophotometer

Figure 4 designates the cross-section of the vacuum chamber of the spectrophotometer. The base of the chamber is a massive plate (2) with a diameter of 450 mm and a thickness of 35 mm. It is equipped with a chamber housing (1) with welded connecting pipes. The camera body is covered by a lid, the presence of which allows to adjust and install the camera. The microcryogenic machine (3) is located at the bottom of the chamber. At the top and body of the cover, there are windows (4) for the introduction of laser interferometer radiation, for measuring the growth rate and controlling the thickness of the sample, as well as for the introduction of globe radiation in the IR range. All vacuum seals of metal prefabricated parts are sealed with copper seals, and salt optics are sealed with Indian seals.

Particularly in Figure 4, by using a magnetic drive (10), it is possible to install and remove a KBr (6) plate on the substrate, the purpose of which is to overlap the work surface (7) with uncontrolled pre-condensation processes and protect it from secondary processes during heating. The distance between the back and the protective panel is 5 mm. Furthermore, additional copper plates (8) are placed on the low-temperature flange of the micro-refrigerator, which acts as a cryocondensation pump. The protective housing (12) has holes that can be overlapped with the magnetic drive (10) of the cylindrical valve (9) for suction of the cryopump. This increases the basic vacuum level in the chamber. In addition, combining the substrate with a protective film, its overlap with a protective plate creates a closed volume near the substrate, the pressure of which is much lower than the external pressure in the vacuum chamber [9].

The matrix gas treatment system includes additional units of the injection system, the scheme of which is illustrated in Figure 5 [10].



1 — test gas “1”, 2 — test gas “2”, 3 — valves, 4 — baratrons, 5, 6, 7, 8 — taps.

Figure 5. Schematic of the cryodensor vapor extraction system

The temperature of the substrate is measured in thermocouples (Au+0.07 % Fe) — Cu, with an accuracy of at least 0.5 degrees in the lower temperature range; IR spectrometer frequency range 400 cm^{-1}÷4200 cm^{-1} (ICS-29); film thickness — 30 microns (two-beam laser interferometer) [11–13].

As an example, interferons obtained during growth in the substrate at a temperature of ethanol condensate in the substance T = 16 K and a gas phase pressure P=8×10–^{4} Pa (Figure 6).

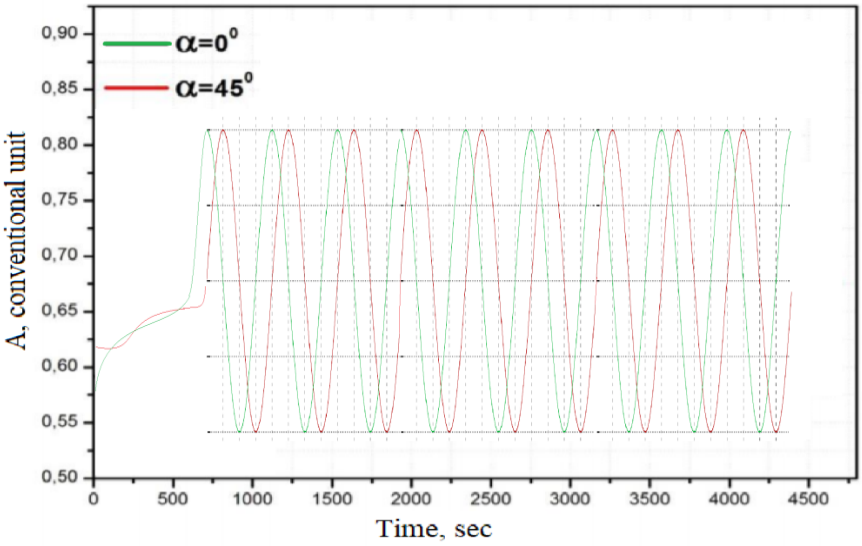


Figure 6. Typical interferograms of condensate film growth

Data registration was performed automatically every 0.5 seconds. The main sources of measurement error are the error in determining the time (interference period) and the angle of incidence. During the alignment of the installation, the angle of incidence of the interferometer was set with an error not exceeding 0.5 %. The error in determining the growth time of the film does not exceed 4.5 % [14–15].

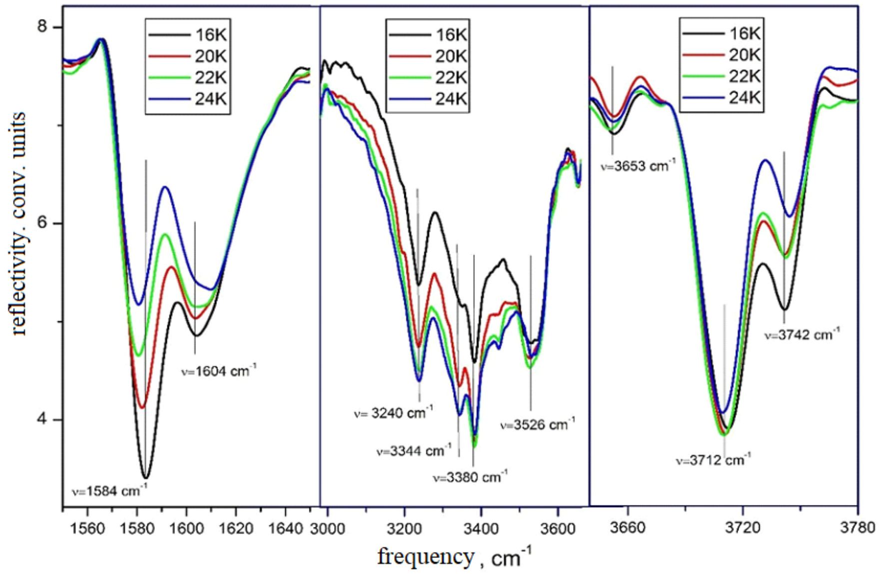


Figure 7. Reflection spectra of water in a nitrogen matrix

In the Fig. 7 the spectra of reflections of the samples in the temperature range of the matrix 16–24 K are given. These are presented in the frequency range of deformation oscillations of water (left drawing), hydrogen-bound states (central drawing) and quasi-free valence symmetric and asymmetric oscillations (right drawing). Similar data are provided in Fig. 8 for the temperature range 26–32 K.

As can be seen from the presented data, the reflection spectra have features specific to matrix-isolated states of water. In the frequency range of deformation curves (left pattern) there are two strips of absorption with a maximum of ν = 1584 cm — 1 and ν = 1604 cm — 1. The first band refers to the deformation oscillations of H_2O monomers in solid nitrogen. The band with a maximum of ν = 1604 cm — 1 can be related to the amount of water polymers in the matrix. In this case, of course, with the increase in temperature of the substrate, the monotonous decrease in the amplitude of the oscillations of the monomers is accompanied by the growth rate of absorption of the polymer.

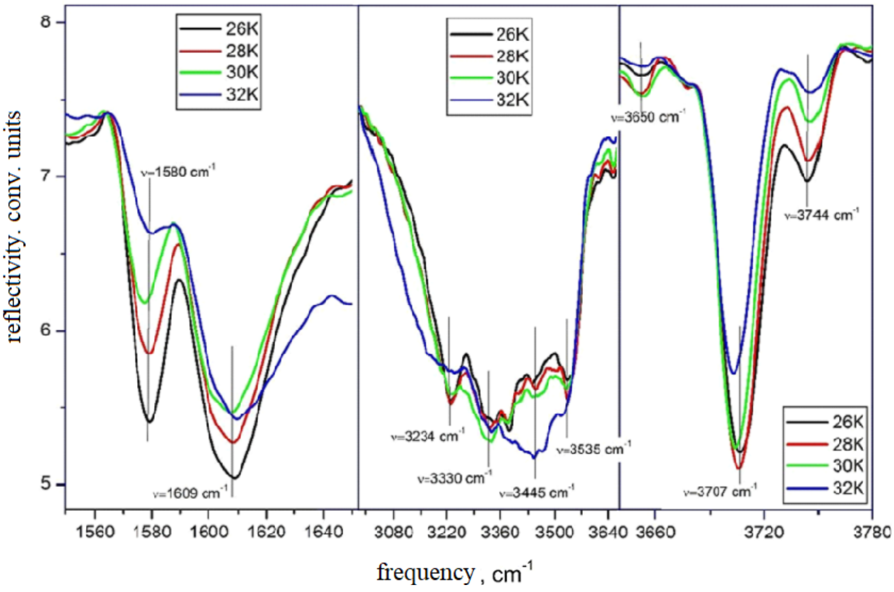


Figure 8. Reflection spectra of water in a nitrogen matrix

The matrix temperature range (Figure 8) is from 26 K to 32 K in the frequency ranges of bending vibrations (left figure), hydrogen-bonded states (central figure), and quasi-free stretching symmetric and asymmetric vibrations (right figure).

The central fragments of Fig. 7 and 8 represent the frequency range of hydrogen-bonded O–H bonds. The spectra given in this range and their agreement with these allow to make assumptions about the availability of polyaggregates of different scales. Thus, the minimum at a frequency of 3234 cm — 1 corresponds to the square meters, the absorption at a frequency of 3330 cm — 1 corresponds to the polymer, and the peak at a frequency of 3526 cm — 1 can relate to the dimers [16]. We do not plan to analyze in detail the cluster composition of the model in this work. It is important to note that the increase in the temperature of the matrix at low temperatures leads to the transformation of spectra in this frequency range.

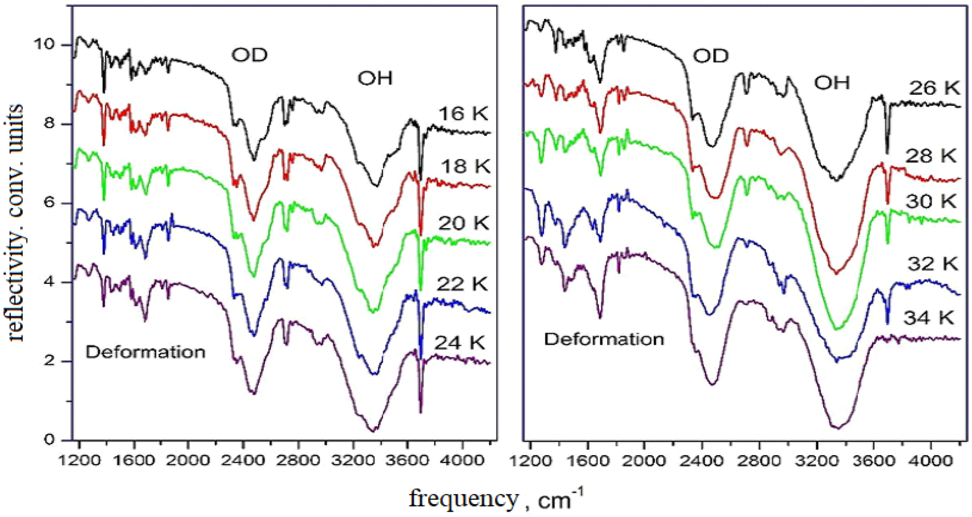


Figure 9. Reflection spectra of an isotopic mixture of water (3 %)
in a nitrogen cryomatrix at different substrate temperatures

Figure 9 demonstrates data in the frequency range of bending and stretching vibrations of the O–H and O–D bonds. As can be seen from the figure, the vibrational spectra contain features characteristic of matrix-isolated systems, in which aggregates of water and heavy water of various sizes are present. A gradual increase in the temperature of the sample leads to a transformation of the spectrum, but the degree of these transformations is different for different types of vibrations of molecules of the isotopic mixture of water.

Conclusions

In accordance with our data, it can be argued that the temperature of the transition from glassy amorphous ice to the state of a superviscous liquid is Tg = 137 K ± 2 degrees. A further increase in temperature leads to a stepwise transformation in the layer. This may be associated with competing crystallization processes through the growth of cubic and hexagonal nuclei, as well as direct crystallization of liquid superviscous water formed at Tg and existing together with the crystalline phase up to temperatures of about 200 K.

Our studies revealed the anomalous behavior of the samples at temperatures preceding sublimation. This is the abrupt behavior of the heating curve, which is accompanied by an extremum pressure in the chamber. In our opinion, these experimental data confirm the point of view expressed in the works [17]. The point is that a multicomponent system consisting of amorphous and crystalline components at a fixed temperature should have different equilibrium pressures of the gas phase corresponding to the partial activation energies of sublimation. Because if the activation energy of the amorphous form of ice is greater than the corresponding values for crystalline modifications, this will lead to the fact that at high temperatures amorphous water will evaporate at an earlier stage, and then recondense on crystalline components into a crystalline form.

Acknowledgments

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А.У. Алдияров, Х.И. Бейсенов, Ұ.П. Сүйінжанова, Д.Е. Ережеп

Полиаморфты өзгерістерді судың криовакумды конденсаттарындағы нитрогенді криоматрицаларын зерттеу

Конденсацияланған заттың қазіргі заманғы физикасының маңызды міндеттерінің бірі – түзілу шарттары мен нәтижесінде пайда болған қатты фазаның қасиеттері арасында бірмәнді байланыс орнату. Оның шешімі қажетті қасиеттері бар материалдарды жасаудағы үлкен жетістіктерге ықпал етеді. Кез келген ғылыми-техникалық мәселелер сияқты, бұл тәсіл де кең ауқымды сұрақтарды шешу қажеттілігімен байланысты. Бұл бағыттағы табыстың негізі кешенді тек модельдік сынақтармен жүзеге асыру болып табылады, егер зерттелетін зат практикалық тұрғыдан маңызды болса, бірақ өзі қызықты физикалық қасиеттерге ие болса; мұндай объектілерді химиялық қасиеттерге толық жатқызуға болады. Сутегі байланысқан заттар, оларда ван-дер-Ваальс күштерінен басқа, молекулааралық сутегі байланысының болуына байланысты өзара әрекеттесу маңызды рөл атқарады. Алынған криоматриксті оқшаулау әдісі адсорбцияланған қабаттағы аралық сатыдағы су мен этанолдың таза компоненттерін криоконденсациялау процесінде сұйық күйге ұқсас қысқа диапазонды кластерлер түзілу процесі жүреді деген болжам жасайды.

Кілт сөздер: кристалдану, қоспалар, әйнектің ауысу динамикасы, кристалл жасушасы, криоконденсация.

А.У. Алдияров, Х.И. Бейсенов, Ұ.П. Сүйінжанова, Д.Е. Ережеп

Исследование полиаморфных превращений в криоматрице азота в криовакуумных конденсатах воды

Одна из важных задач современной физики конденсированного состояния — установить однозначную связь между условиями образования и свойствами образующейся твердой фазы. Его решение будет способствовать крупному прорыву в создании материалов с заданными свойствами. Как и любая научно-техническая проблема, такой подход связан с необходимостью решения широкого круга фундаментальных проблем. Основой успеха в этом направлении является выполнение комплекса не только модельных испытаний, когда исследуемое вещество важно с практической точки зрения, но само по себе имеет интересные физические свойства. К таким объектам в полной мере можно отнести химические свойства. Вещества с водородными связями, в которых, помимо сил Ван-дер-Ваальса, важную роль играют взаимодействия, обусловленные наличием межмолекулярной водородной связи. Полученный метод криометрического выделения делает предположение, что в процессе криоконденсации чистых компонентов воды и этанола на промежуточной стадии в процессе адсорбции происходит образование кластеров, сосуществующих друг с другом.

Ключевые слова: кристаллизация, смеси, динамика стеклования, кристаллическая ячейка, процесс криоконденсации.

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